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NON DESTRUCTIVE EXAMINATION OF NON METALLIC MATERIALS - RESEARCH NEEDS AND OPPORTUNITIES

Report No. 88-2-K

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ABSTRACT

A review of the current status of non destructive examination (NDE) of non metallic materials was carried out to ascertain the possibilities for applications within the Materials Chemistry Section. The review has a dual focus to identify both the more promising application domains amenable to NDE and the techniques likely to be of assistance in the evaluation/ characterization/assessment of non metallics, initially in a laboratory setting, but ultimately for field application. The necessary steps to develop an NDE program with due regard for existing expertise elsewhere in the Division are outlined.

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EXECUTIVE SUMMARY

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A review of the current status of non destructive examination (NDE) of non metallic materials was carried out to ascertain the possibilities for applications within the Materials Chemistry Section. The review has a dual focus to identify both the more promising application domains amenable to NDE and the techniques likely to be of assistance in the evaluation/ characterization/ assessment of non metallics, initially in a laboratory setting, but ultimately for field application. The representative applications include adhesion, (as encountered in sealants or coatings), strength of reinforced rubber (flexible seals) and reinforced plastic (FRP piping) and control of curing systems (radioactive waste forms). The recommended NDE techniques include thermography, ultrasonic inspection and radiography. Other more narrow techniques which could see limited applicability would be acoustic emission, microwave, and holographic interferometry. The necessary steps to develop an NDE program with due regard for existing expertise elsewhere in the Division are outlined.

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To: Dr. D. Mills
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NON DESTRUCTIVE EXAMINATION OF NON METALLIC MATERIALS - RESEARCH NEEDS AND OPPORTUNITIES

J.M. Braun

1.0 INTRODUCTION

Utilities such as Ontario Hydro have a large incentive in maintaining an orderly planned materials program to ensure timely inspection and replacement of critical plant equipment components. Large sums of money are invested in research programs prior to start up or commissioning of units to ascertain the likely performance expectations and derive appropriate replacement schedules. Much additional information can be gained by periodic inspection of the components of interest to detect accelerated degradation or incipient failures.

Non destructive examination (NDE) methods have been widely used in this area to assess initial quality/conformity to the manufacturing specifications and detect service induced changes and/or defects. Commercial, dedicated instrumentation is readily available to perform these tasks, using a number of different instrumental techniques. However applications so far have been largely restricted to critical metallic components, presumably because of the consequences of unexpected failures. This approach has also been applied to non metallics but only to a very small extent and then mostly to laminates and particularly to those used in the aircraft industry where unexpected failures are critical. Considerable savings could be realized if the same approach could be extended to a broader class of non metallics components such as used by Ontario Hydro. Current NDE capabilities as practiced during field inspections rely mostly on a visual inspection augmented by hardness measurements. Much interest and thought has been generated in the Materials Chemistry Section - the present report being one such contribution - in trying to assemble an effective program/1,2/.

Non metallic materials represent a major challenge in the practice of NDE for a number of reasons. Unlike metals they do not exhibit a well defined morphology and the concept of "defect" must be applied more loosely. Furthermore relatively small, often undetectable changes in chemical structure (ie crosslinking, chain scission, oxidation) can bring about considerable changes in physical properties and performance. It is thus unclear at the outset which physical material properties would be most appropriate to follow and gain the insights necessary to understand and characterize aging and/or failure. Finally the lesser density of the materials and their physical characteristics result in poorer performance of the most effective conventional NDE tools: eddy current (dielectric), radiography (contrast) and ultrasonics (poor penetration).

Great strides have however been made in the characterization of specialized non metallic components: typical examples would include the detection of partial discharge activity of energized electrical insulators to detect the existence of minute voids in the bulk of the material and laser detection of impurities in the extrudate melt of high voltage cables. It is readily apparent though that such developments cannot be readily implemented for other components even where the existence of voids in large casting would be equally critical for lack of the necessary voltage withstand. Clearly a generic approach to NDE of non metallics is required and this would be even more beneficial where successful implementations could be readily transferred to other materials and situations.

The present report presents an overview of the current status of non destructive examination of non metallic materials, the possibilities for applications within the Materials Chemistry Section and the necessary steps to develop an NDE program with due regard for existing expertise and efforts elsewhere in the Division. The review will have a dual focus to identify both the more promising application domains in the Section and the techniques likely to be of assistance in the evaluation/ characterization/assessment of non metallics, initially in a laboratory setting, but ultimately for field application. It is realized at the outset that the two sets, techniques and applications, may not always overlap and that the bulk of the research is usually carried out on systems that are amenable to study and not necessarily the more relevant ones to our applications.

2.0 POTENTIAL NDE APPLICATIONS

The activities of the Materials Chemistry Section are extremely diverse, covering long term research investigations on the performance of non metallics in plant equipment and radioactive waste disposal, shorter term studies, field inspections and routine testing of paints, dielectric and hydraulic fluids. The properties of interest are also very diverse and can include

mechanical strength (fiber reinforced plastics, seals), electrical performance (SF_6 spacers, cables), permeation (tritium through protective clothing), leaching (radioactive wastes) and the effects of aging on these properties. While it is inconceivable to find a generic technique to address all of the above, groupings of applications might be possible, concentrating on only a few powerful techniques.

Non destructive evaluation techniques have been developed in narrow areas, whether as SF_6 byproduct detector or as tagging compounds for turbogenerators, and these activities are expected to continue: there is currently much effort in trying to characterize the aging factors of SF_6 spacers and in particular the nature of the gases held in the voids. By their nature however these activities are excluded from the purview of this study because of their specificity.

Deficiencies in either new or aged materials can often be readily detected, but their impact on actual performance is considerably more difficult to evaluate. Because of their unique time dependent properties and the rather large free volume reducing molecular packing densities, plastics and rubbers exhibit failure mechanism vastly different from those encountered with other materials, notably metals and ceramics. In particular the concept of flaw nucleation and propagation to failure has not proven a fruitful one in this particular area and modeling efforts are virtually non existent. The case is further complicated by the use of many and diverse additives, whether filler, processing aids, reinforcements, antioxidants, etc in the final formulation to confer the desired properties to the finished product. In their unreinforced neat form the properties of polymers are largely determined by their transition temperatures, most importantly the glass transition (T_g) and the melting point, but also sub T_g transitions involving local chain mobility. Aging and the attendant changes in the chemical structure of the polymeric networks affect these transitions and thus the failure mechanisms.

A careful analysis for each application is required to establish the critical material properties and the possible effects of the environment on long term durability. An example will serve to illustrate this point very readily: in gas insulated switchgear massive epoxy spacers are used as electrical insulators and these are tested stringently to reject any castings with voids larger than about 500 um at the highest voltage levels as these could sustain partial discharges leading ultimately to failure. In any other but electrical applications such voids would be of no concern, in effect it has been shown that they could improve fracture toughness. Much research effort has been devoted to the fine characterization of the structure, properties and their interrelationship in non metallic materials both in solution and in the solid state. While some of the techniques commonly used in other materials areas could be readily applied, eg X-ray

diffraction and all forms of microscopy and spectroscopies, a good deal of the research was devoted to developing a better understanding of the molecular entanglements and the glassy state, with developments such as gel permeation chromatography and NMR magic angle spinning to enable investigations of solids. However in spite of the detailed molecular understanding gathered over the years, the gap between the atomic level and ultimate properties (mechanical, adhesion, high temperature, electrical, ...) has been only partially bridged: chemical changes on aging can hardly ever be used to predict current performance and remaining life and destructive tests are routinely used. The challenge, and it is obviously a formidable one, in applying non destructive tests for the evaluation of non metallics will be to find these techniques capable of providing information closer to the end use properties more so than at the molecular level.

For the purposes of the present review, applications are best divided into quality control type tests involving new materials and performance assessment at some point during the life of the material. Furthermore these can in turn be subdivided into major applications' groups: these would include adhesion to a substrate, cure development, mechanical and electrical strength. Adhesion applications include paints, sealants, roofing membranes, and obviously all adhesive fastening such as practiced in the Pickering NGS vacuum building to reinforce fiberglass risers and downcomers. Of particular concern here is the strength of the bond both initially when formed and as affected over time by its environment. While it is known that the interface can be chemically affected, bond strength in effect can range anywhere from 100% down to bare contact without any strength: quantification of the bond strength and changes with time would be the primary parameter.

A unique application represents the solidification of low and medium level wastes: radioactive solids and liquids dispersed in a number of liquid matrices (polyester, epoxy, cement, etc) and these are cured to a solid. The prime parameter here is the assurance of initial integrity evidenced by full cure and freedom from liquids and cracks in the final product. It is envisaged that the solidification would be performed in a 200 L steel or plastic drum: the chosen technique(s) must be able to perform under these conditions.

Strength is the critical factor in most applications involving reinforced plastics and particularly rubbers. In the latter case the rubber provides a flexible protective matrix for the fibrous strength member of the composites. Much the same is achieved with fiberglass reinforced plastic where the plastic matrix provides the chemical resistance and the fibers the strength. Typical examples include various seals (roof seal, pressure relief duct seals, valve diaphragms,...), conveyor belts, hoses and in the case of FRP mostly piping and electrical insulators. Of interest then are initial strength or any variant thereof, eg

modulus of elasticity/stiffness and slow deterioration with time under a number of environments, thermal, radiation,... A specific example would include the recent delivery of FRP manhole covers for the Bruce A NGS vacuum building dousing system which were found to have a systematic construction/design defect: the problem was uncovered (destructively) as a result of additional testing carried out as part of an aging program.

It should be recognized also that many non metallic material applications, while appearing to relate to properties such as electrical performance, are more critically affected by their mechanical properties and this will be the normal failure or life limiting mode. Examples include most low voltage wiring applications, whether in residential wiring systems or as nuclear control cables, and thermal insulation applications. Wiring performance, particularly for control cables includes a stringent thermal aging requirement with end of life determined by the point at which the insulation specimen substantially looses its mechanical properties and can no longer sustain bending.

In reviewing the broad range of applications for non metallic materials encountered in the Materials Chemistry Section it becomes readily apparent that there are numerous critical properties that in any one case can become the overriding material parameter. In effect the major challenge in determining the performance of these materials is to achieve a comprehensive listing of critical parameters and their likely boundary: this serious limitation has by necessity limited the extent and value of generic research in furthering our understanding of non metallics. These limitations will also apply to the study of key properties by non destructive evaluation techniques. Only by careful review of the potential NDE techniques and how these relate to potential application can we expect to make any progress.

3.0 POTENTIAL NDE TECHNIQUES

The research and development activities on NDE techniques are considerable including a substantial effort at the Ontario Hydro Research Division. However the effort devoted to non metallics is very limited in comparison to the research on metals; if one were to exclude all research on fiber reinforced plastics and their aerospace applications, the effort is almost insignificant.

A few groups have started to devote resources to non metallics, often as a spinoff from conventional NDE activities and mostly in narrow and specialized areas/3,4/. Contacts were made with many of these groups and these proved invaluable in conveying a critical perspective on much of the published literature. The most active groups in Canada include NRC's IMRI and NAE, U of T, Queen's and the New Brunswick Research and Productivity Council. Close contact should be maintained with these groups as our

effort should be integrated with the capabilities and expertise available in the Division, most particularly in the Non Destructive and Fracture Evaluation Section.

In reviewing potential techniques, certain approaches such as eddy current inspection and magnetic particle inspection which are only marginally applicable to non metallics were not included here: capabilities are well established, here or elsewhere, such as for the evaluation of (steel reinforced) conveyor belts/5/. The non destructive evaluation techniques presented in the next sections were divided for convenience into electromagnetic and mechanical techniques.

3.1 Electromagnetic Techniques

3.1.1 Radiography

Radiography is probably the oldest of NDE techniques applied to non metallics. It is based on the differential absorption of a penetrating radiation, typically X or gamma rays with good resolution at the short wave lengths. Typically this is recorded on film or other detector for further image manipulation and enhanced resolution: in its most advanced implementation three dimensional tomography can be performed fairly readily. Based on differential absorption, the detection of voids, inclusions and disbonds can be made/6/. Commercial instrumentation including portable systems for field use are readily available.

This technique is currently under active investigation by the Materials Chemistry Section and the subject of a separate report/7/. As such it will not be discussed further here.

3.1.2 Optical Interference Techniques

Optical interference techniques are not properly speaking NDE techniques but rather the measuring back end of other techniques: they record the optical interference fringes created on a surface as a result of an applied stress and compare this to the unstressed state of the material. The stresses can be of mechanical, thermal or vibrational origin. Changes (defects) in the bulk of the material can be detected if this results in a surface displacement under stress. Typical techniques include holographic interferometry, laser speckle photography and moire interferometry/8/.

Typical applications include the visualisation of high stress areas in pressurized vessels and stressed composite components or the detection of disbond areas in tires and or adhesives: weaker areas give rise to higher surface strains and their detection by interference/3/. Other applications are to be discussed in subsequent sections on other techniques for which they represent the "detector".

Capabilities have been developed in the Research Division to perform laser holography and where appropriate collaboration with other groups should be continued.

3.1.3 Thermography

Thermography is probably the technique that is currently the most investigated for applications to non metallics and which holds great promise. Thermography involves the application of heat, the measurement of the resultant thermal field by an infrared detector and manipulation of the resultant image to gain maximum resolution/9,10/. The temperature can result either from internal heat generation, eg from mechanical or vibrational loading, or directly from heat sources such as lasers or focussed heat lamps/11,12/. Bulk characteristics of the material are again deduced from their effect on the surface thermal field and the associated (bulk) thermal diffusion. By the appropriate manipulation of the optics, the size of the field to be investigated may range from microscopic sizes to areas of several square meters. Whenever the resultant temperature leads to a surface displacement, the effects can then also be determined by an interferometric technique/13/.

As is readily obvious, thermography involves supramolecular, morphology related properties, notably heat transfer characteristics and as such is most attractive in bringing information on engineering properties. Changes in density, disbonds, or voids can be detected. The techniques have been used extensively, for instance to map defects in graphite epoxy, to find disbonds in sheet molding compounds and coatings, interlaminar flaws in composites, etc/14-17/.

Commercial instrumentation, though sophisticated to provide high temperature and spatial resolution, is readily available. As direct (versus mechanically induced) thermographic methods do not require physical contact, excellent potential for field applications exists.

3.1.4 Microwave Inspection

Microwave NDE involves the use of electromagnetic radiation in the 1 to 500 GHz range: both physical effects (transmission and reflection/refraction) and molecular absorption of the material are measured. As the energy is in the range of the chemical bond energy of organic materials, changes in the chemical composition such as induced by polymerization, cure, water absorption and oxidation will be detected. Discontinuities of the order of the wavelength (0.1 to 1.0 cm) may also be detected with good penetration into the material.

The potential of microwaves as an NDE technique for non metallics was recognized as early as the 1960's but was not developed,

except for some highly specific applications, particularly for large rocket propellant casings/18-20/. It is believed that the lack of interest in the technique is mostly due to the nature of the information provided: either too gross for defect detection or as a spectroscopic technique to yield molecular information comparable to what can be obtained by other techniques whether IR or NMR but generally unrelated to ultimate physical properties. In specific instances, particularly for the measurement of water content in paper manufacture or where the detailed chemistry of thick samples is of interest, this technique could afford sizable benefits/21/; as a generic tool however it is not seen as one of the more powerful techniques.

3.1.5 Magnetic Resonance Techniques

Nuclear magnetic and electron spin resonance (NMR, ESR) are powerful spectroscopic techniques used to provide chemical bonding information. Because of relaxation times and associated signal broadening NMR is mostly applicable to liquids but through artifacts (eg magic angle spinning) has been applied to solids. Certain solids such as the human body have large amounts of free liquids and this has led to the use of NMR as a nondestructive imaging technique, particularly in the medical field. The method can be used similarly for the mapping of absorbed fluids in polymers and composite materials/22/. Much as microwave techniques NMR could be successful in specific applications (eg mapping water trees in electrical cable insulations) but not as a generic NDE technique for non metallics. ESR offers somewhat different capabilities related to the formation of free radicals during mechanical damage: limited attempts have thus been made to characterize the remaining life of nylon ropes/23/. The technique is however to specialized to become one of the generic techniques we are trying to develop.

3.2 Mechanical Techniques

3.2.1 Ultrasonic NDE

Ultrasonic NDE one of the more prevalent NDE technique and has been applied quite extensively to non metallics. Ultrasonic NDE involves the interaction of high frequency sound waves with the material and, in its simplest form, the measurement of either the reflected or transmitted signal. The method is sensitive to defects, delaminations (interfaces) and certain material changes and these can be determined for a point (A-scan), line (B-scan) or plane (C-scan). As ultrasound attenuates rapidly in air, a couplant medium is required: a gel, oil, water or more recently dry couplants. Alternatively a high power laser beam can be used to create ultrasonic waves.

The technique has found extensive use for FRP composites, mostly in the aerospace industry and is also starting to penetrate other

areas/24-26/. It has also been used in tire/27,28/ and polyethylene joint/29/ inspections, detection of cure, chain orientation, crystallinity, moisture absorption, measurement of elastic constants, etc/29-34/. As the travelling mechanical waves are modulated by the morphology of the material it has potential to measure engineering properties, including strength and fatigue characteristics.

It should be recognized, however, that ultrasonic characterization of non metallics such as plastics and rubbers is radically different from that of metallics, both in hardware and in approach. The critical differences in the two areas are a result of the much stronger signal attenuation in non metallics and its sharp frequency dependence: at the higher frequency (>20 MHz) there is virtually no penetration. Yet these high frequencies are the ones sought after to achieve high resolution and are most commonly used with metals and ceramics because of the nature of the defects of interest. Furthermore there is essentially no signal reflection and associated signal analysis, a fertile area of research in metals. In recognition of the special needs of non metallics there a slow realization that the existing hardware and thrust towards ever higher resolution are inappropriate: suppliers are starting to offer high energy, low frequency, broad band pulsers. New developments include dry coupling techniques and non contact laser generation and detection of ultrasounds/35-37/.

By examining the frequency and temperature dependence of the ultrasonic signal additional information on material strength can be obtained either via acousto-ultrasonic or by a form of mechanical spectroscopy: specific applications include the measurement of adhesive bond strength and fatigue damage in composites/30/.

The ultrasonic technique is seen as providing one of the more powerful NDE tools, particularly as instrumentation is readily available and generally inexpensive. It is readily clear also that the special needs of the non metallic materials must be recognized both in the instrumentation chosen and the properties investigated: despite a strong research effort in the area neither are presently available and these should be developed.

3.2.2 Acoustic Emission

Acoustic emission (AE) is referring to the measurement and quantification of stress waves generated by the release of strain energy (in the acoustic range) that occurs during crack growth or other material deformation. Unlike other techniques discussed here, the materials must be loaded, presumably below a safe level, to produce acoustic emission and the technique is not absolutely non destructive/38,39/.

Acoustic emission inspection has been used extensively for inspection of fiber reinforced composite materials, in particular at Ontario Hydro for FRP booms, and is credited with a significant reduction of corrosion resistant FRP vessel failures. The analysis of acoustic emission signals is quite complex but the technique is very attractive as the entire sample under investigation (barring AE propagation) can be tested. While the technique is used foremost for FRP material, it has also been used to detect curing in epoxies and for the evaluation of rubbers, a notorious acoustic emission absorber/40/. A particularly elegant implementation has seen the use of embedded optical fiber wave guides as part of the composite structure.

Acoustic emission inspection equipment is readily portable and is well suited to field applications. While it is far too restrictive for the range of applications considered, it should nonetheless be considered as potential technique in specific cases. Given the sophistication of instrumentation available at the Research Division, exploratory studies could be readily carried out in collaboration with Metallurgical Research and these avenues should be pursued.

4.0 STRATEGY AND PROGRAM

The thrust of the Materials Chemistry Section NDE program is to improve capabilities in the characterization of non metallics and especially of their ultimate/engineering properties. As such it should complement our efforts dedicated to bettering our understanding of the aging characteristics and attendant molecular changes taking place in the materials and where destructive techniques can play a dominant role. In view of the diversity of applications encountered it is proposed that the program be limited, at least initially, to a few representative applications and the more promising NDE techniques. At the same time it is envisaged that selected NDE techniques, which do not seem to offer the broad range sought for generic developments, could be further investigated for specific applications, on a project basis with either internal or external collaboration to minimize capital expenditures.

The representative applications are seen to include the following: adhesion (as encountered in sealants or coatings), reinforced rubber (flexible seal), reinforced plastic (FRP piping), curing system (radioactive waste form). In addition to radiography the NDE techniques should be comprised of thermography and ultrasonic inspection. The more narrow techniques which could see limited applicability would include acoustic emission, microwave, and holographic interferometry. It is particularly encouraging to find that both techniques considered for full investigation have the breadth to be relevant and/or have been used previously in related investigations over the entire range of applications considered.

It is quite clear at this time that a detailed program of NDE activities is not yet appropriate: major efforts are going to be required in 1988 in the preparation of adequate specifications for the three prime techniques, their commissioning and preliminary experimental work. It is also quite clear from the experience elsewhere that the NDE of non metallics is a significant technical challenge requiring a multi year commitment of resources and capital expenditures. It is estimated that given the diversity of applications tackled a minimum annual staff commitment of approximately 2 000 professional and 1 000 technical hours would be required. It is further estimated that a comparable effort be maintained over a period three years to see successful applications implemented with confidence under field conditions. Part of this commitment, possibly 20% of it, could be devoted in support of exploratory work on the more specific techniques: it is envisaged that the bulk of this work be undertaken as a collaborative effort, internally or externally as the situation warrants.

It is foreseen that the three techniques would follow much the same development path: reference samples materials representative of the selected applications would be manufactured and controlled imperfections - be they defects, over or undercures, ... - introduced to assess the ability of the technique to detect such changes. In a second phase of the work finer, aging related material changes would be brought about by accelerated aging procedures and the techniques reassessed. As a better understanding of each technique is gained, and there will no doubt be vast differences in the time required to achieve this, a fuller range of applications can be drafted and incorporated into a comprehensive program.

In view of probable developments elsewhere, it is fully recognized that our initial expectations and or selected techniques may need to be reconsidered: it is particularly important that the very useful contacts made so far in the evaluation phase be maintained and broadened where applicable to allow us to make the necessary changes in our programs.

5.0 CONCLUSIONS AND RECOMMENDATIONS

A review of the current status of non destructive examination (NDE) of non metallic materials was carried out. The thrust of the review is to improve capabilities in the characterization of non metallics and especially of their ultimate/engineering properties, complementing our understanding of the aging characteristics of materials. The representative applications include adhesion properties (as encountered in sealants or coatings), strength of reinforced rubber (flexible seals) and reinforced plastic (FRP piping) and control of curing systems (radioactive waste forms).

The recommended NDE techniques include thermography, ultrasonic inspection and radiography. Other more narrow techniques which could see limited applicability would be acoustic emission, microwave, and holographic interferometry. An annual staff commitment of approximately 2 000 professional and 1 000 technical hours over a three year period is recommended. Part of this commitment, possibly 20% of it, could be devoted in support of exploratory work on the more narrow techniques. A strong emphasis on collaborative effort whether internal or external should be maintained throughout the program.

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